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Pitting Corrosion of $\text{Ni}_3(\text{Si,Ti})+2\text{Cr}$ Intermetallic Compound at Various Chloride Concentrations

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Abstract - The pitting corrosion of $\text{Ni}_3(\text{Si,Ti})$ with 2 at% Cr containing two regions of a $\text{Ni}_3(\text{Si,Ti})$ single-phase of L_{12} structure and a mixture phase of ($\text{L}_{12} + \text{Ni}_{ss}$) was investigated as function of chloride concentrations by using a polarization method, scanning electron microscope and energy dispersive X-Ray spectroscopy in neutral sodium chloride solutions at 293 K. The pitting corrosion of $\text{Ni}_3(\text{Si,Ti})$ with and without the addition of aluminium and type C276 alloy were also studied under the same experimental condition for the comparison. The pitting potential obtained for the $\text{Ni}_3(\text{Si,Ti})$ with 2 at% Cr decreased with increasing chloride concentration. The specific pitting potentials and the pitting potentials were decreased in the order of C276 alloy > $\text{Ni}_3(\text{Si,Ti})$ > $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$ > $\text{Ni}_3(\text{Si,Ti}) + 4\text{Al}$, which means that the pitting corrosion resistance of $\text{Ni}_3(\text{Si,Ti})$ with 2 at% Cr was higher than $\text{Ni}_3(\text{Si,Ti})$ with 4 at% Al, but lower than that of $\text{Ni}_3(\text{Si,Ti})$. A critical chloride concentration of $\text{Ni}_3(\text{Si,Ti})$ with 2 at% Cr was found to be higher than that of $\text{Ni}_3(\text{Si,Ti})$ with at% Al. In addition, the presence of high concentration for oxygen indicates the occurrence of pit formation.

Keywords : Intermetallic Compound; Pitting Corrosion; Sodium Chloride; Polarization, Scanning Electron Microscope, Energy Dispersive X-Ray Spectroscopy

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I. INTRODUCTION

The nickel base intermetallic compounds such as $\text{Ni}_3(\text{Si,Ti})$ with and without addition of chromium (Cr), showing a high melting temperature, have advantages of lightweight and high-temperature oxidation resistance compared with conventional alloys such as steel and nickel-based alloys (Takasugi et al., 1990 ; Kaneno et al., 2011). Therefore, those intermetallic compounds could be applied as high-temperature materials in the future (Takasugi et al., 2002). On the other hand, an improvement in the oxidation resistance of the $\text{Ni}_3(\text{Si,Ti})$ intermetallic compound was attempted with macro-alloying by adding Cr, which enhanced that resistance at high temperatures (Kaneno et al., 2011).

Furthermore, $\text{Ni}_3(\text{Si,Ti})$ with 2 at.% Cr were composed of two regions of the L_{12} single-phase and a two phase mixture of L_{12} and Ni_{ss} , where Ni_{ss} was a fcc Ni solid solution as well as the $\text{Ni}_3(\text{Si,Ti})$ with Mo and Al (Kaneno

et al., 2011). In addition, it was also found that the addition Cr was slightly effective in improving high-temperature ductility property (Fujimoto et al., 2011). On the $\text{Ni}_3(\text{Si,Ti})$ with 2 at.% Cr, before conducting polarization test, Gadang and co-workers also found that the main element of Ni was present uniformly (Priyotomo et al., 2012). On this compound, Cr was enriched in the two phase region of L_{12} and Ni_{ss} , while Ti and Si are enriched in the L_{12} single-phase region (Priyotomo et al., 2012). However, the $\text{Ni}_3(\text{Si,Ti})$ intermetallic compounds are prone to environmental embrittlement (hydrogen embrittlement) at room temperature in moist environment (Takasugi et al., 1993a,b), where that embrittlement takes place with permeation of atomic hydrogen through electrochemical reaction into the bulk of the compounds.

Up to the present, the intermetallic compound such as $\text{Ni}_3(\text{Si,Ti})$ with the addition of Cr have not systematically

been investigated for the pitting corrosion behavior in neutral chloride concentration. On the other hand, the pitting corrosion behavior of $\text{Ni}_3(\text{Si,Ti})$ with and without addition of Mo and Al already were investigated in neutral chloride solution (Wagle et al., 2011a,b ; Priyotomo., 2013).

Therefore, the aim of this work is to explain the pitting corrosion behavior of $\text{Ni}_3(\text{Si,Ti})$ with addition of 2 at % Cr in various chloride concentrations at 293 K and to compare with that of $\text{Ni}_3(\text{Si,Ti})$ intermetallic compound without and with Al, and C-276 alloy as the references (Priyotomo, 2013 ; Priyotomo et al.,2014).

II. EXPERIMENTAL

A. The specimens

Ni-11 at.% Si- 7.5 at.% Ti-2 at.% Cr compound with the addition of 50 wt. ppm (part per million) of boron (B) was made by using an arc melting method under an argon gas atmosphere in a very high vacuum electric furnace. It was homogenized at 1323K for 48 hours under argon atmosphere and then cooled in the high vacuum furnace with cooling rate of 283 K/ min. C276 alloy, $\text{Ni}_3(\text{Si,Ti})$ compound (Priyotomo et al.,2013) and $\text{Ni}_3(\text{Si,Ti})+4\text{Al}$ compound (Priyotomo et al.,2014) also were as the references for the experiments. The chemical and nominal compositions of materials are shown in Table 1.

Table 1: Composition and nominal of the materials investigated

Elements	$\text{Ni}_3(\text{Si,Ti})$ +2Cr	$\text{Ni}_3(\text{Si,Ti})$ +4Al*	$\text{Ni}_3(\text{Si,Ti})^{**}$	C276**
At%				
C	-	-	-	0.02
Si	11.0	11.0	11.0	0.08
Mn	-	-	-	1.00
P	-	-	-	0.03
S	-	-	-	0.03
Ni	79.5	79.5	79.5	50.99
Cr	2.0	-	-	16.5
Mo	-	-	-	17
V	-	-	-	0.35
Fe	-	-	-	7
Ti	7.5	5.5	9.5	-
W	-	-	-	4.5
Co	-	-	-	2.5
Al	-	4.0	-	-
ppm				
B	50	50	50	-

*(Priyotomo et al.,2014) and **(Priyotomo et al.,2013)

For the microstructure observation, a galvanostatic etching of the mechanically polished specimens was conducted in a solution consisting of 15 ml of $17.8 \text{ kmol/m}^3 \text{ H}_2\text{SO}_4$ and 85 ml of methanol at a current density of 0.446 A/cm^2 for 30 seconds (s) at a temperature of 243 K.

B. Polarization test

The specimens were polished with silica carbide paper up to 1200 grit and then were rinsed with distilled water and cleaned in an acetone ultrasonic bath. The test solutions were neutral sodium chloride solutions with

various chloride concentrations (0.00001 to 1 kmol/m^3), which were made with the distilled water and high grade chemical reagent. The test temperatures used was 293 K. The polarization experiments were carried out in a round bottom cell of 1000 ml capacity using a potentiostat apparatus (Reference 600, Gamry Instrument), interfaced to a personal computer. The potential values were measured by using a reference electrode of a saturated calomel electrode (SCE). All potentials referred in this work are with correspond to SCE for the reference electrode. The surface of the working electrode was covered with epoxy resin to expose a test area of 0.6 cm^2 . A platinum wire served as the counter electrode. Prior to potentiodynamic anodic polarization and potentiostatic polarization tests, the open circuit potential (OCP) was monitored and the experiments were begun after the stabilization of OCP for 1 hour. Furthermore, all the data were duplicated to ensure reproducibility of the results. Those experiments were carried out at a scan rate of 0.167 mV/s . After conducting the experiments, the morphology and distributed elements of the specimen surface were investigated and observed by using scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS).

III. RESULT AND DISCUSSION

A. Microstructure before polarization test

Fig. 1 shows microstructures of $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$ after the final polishing with galvanostatic etching process. It is found that $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$ are composed of L_{12} phase (dark area) and $(\text{L}_{12} + \text{Ni}_{\text{ss}})$ mixture region (white area) as well as those phases in $\text{Ni}_3(\text{Si,Ti})$ with the addition of Al and Cr (Priyotomo et al.,2012). Furthermore, Kaneno and co-workers have already investigated and elucidated the microstructure and the phases of this compound in more detail (Kaneno et al., 2011).

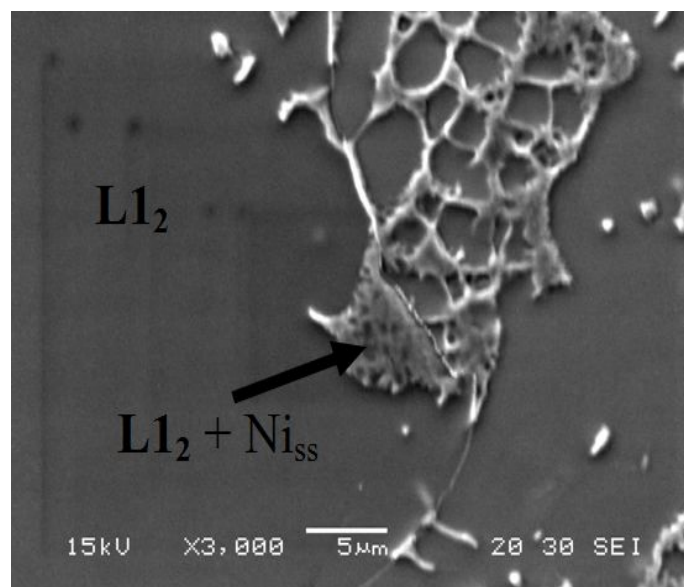


Fig. 1 Microstructure of $\text{Ni}_3(\text{Si,Ti})+2\text{Cr}$ before polarization test

B. Determination of pitting potential (E_{pit})

Fig. 2 shows the potentiodynamic anodic polarization curves for $Ni_3(Si,Ti) + 2Cr$ intermetallic compounds in neutral sodium chloride solutions with various chloride concentrations at 293 K. The anodic current densities of these intermetallic compounds were very small up to a certain applied potentials, which referred to passive current densities. Furthermore, the anodic current density began to increase above a certain potential and suddenly increased with increasing applied potential. Fig. 3 shows a representative figure of a pit appearance for $Ni_3(Si,Ti) + 2Cr$ after polarization test in 0.1 kmol/m³ neutral sodium chloride solution at 293 K by applying scanning electron microscope (SEM) with secondary electron imaging method. The Fig 3 shows that the rapid rise in the anodic current density was caused by the initiation and propagation of the pit with an irregular shape. As shown in the inset figure of Fig. 2, the value of pitting potential (E_{pit}) was measured through the extrapolation of the increased anodic current density to the passive current density. On the other hand, the magnitude of the anodic current densities for $Ni_3(Si,Ti)+2Cr$ and $Ni_3(Si,Ti) + 4Al$ (Priyotomo et al.,2014) were more than one order higher compared to those for $Ni_3(Si,Ti)$ intermetallic compound (Priyotomo., 2013).

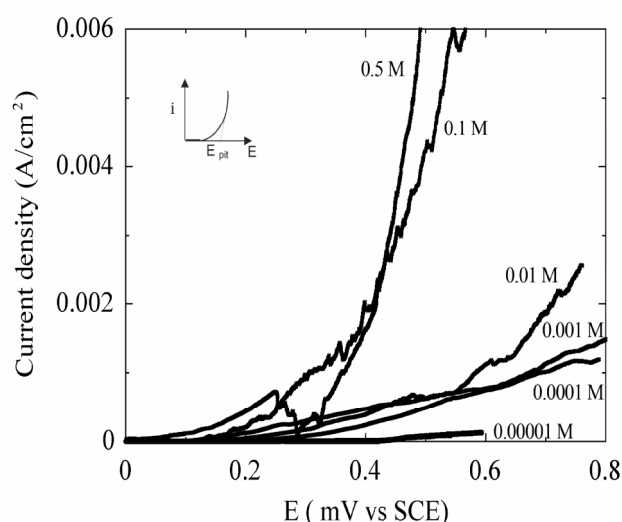


Fig.2. A potentiodynamic anodic polarization curves for $Ni_3(Si,Ti)+2Cr$ intermetallic compounds with various chloride concentrations (kmol/m³) at 293 K.

In this work, no pit was found on the surface of $Ni_3(Si,Ti)+2Cr$ at the chloride concentrations less than 0.0055 kmol/m³ after the measurement of anodic polarization curves as well as the surface of $Ni_3(Si,Ti)+4Al$ at the same condition (Priyotomo et al., 2014). Therefore, it was clearly found that no pitting corrosion took place at the chloride concentration less than 0.0055 kmol/m³ as well as the $Ni_3(Si,Ti)+4Al$ compound (Priyotomo et al., 2014). Furthermore, at a chloride concentration higher than 0.0055 kmol/m³, the pitting potential was measured through the extrapolation method of the rapid increased anodic current density to the passive current density as shown in the figure inserted in Fig. 2, and an apparent pitting potential in the same method was also got at the

chloride concentrations less than 0.0055 kmol/m³ with no occurrence of pitting corrosion (no pit) as well as $Ni_3(Si,Ti) + 4Al$ intermetallic compound in neutral NaCl solution (Priyotomo et al.,2014). In addition, Priyotomo had already found that no pits were found on the surfaces of type C-276 alloy and $Ni_3(Si,Ti)$ intermetallic compound at the chloride concentrations less than 0.05 kmol/m³ and 0.01 kmol/m³, respectively after measurement of anodic polarization curves, whereas at a chloride concentration higher than 0.05 kmol/m³ for the surfaces of C276 alloy and 0.01 kmol/m³ of $Ni_3(Si,Ti)$ intermetallic compound, pits were found clearly (Priyotomo.,2013).

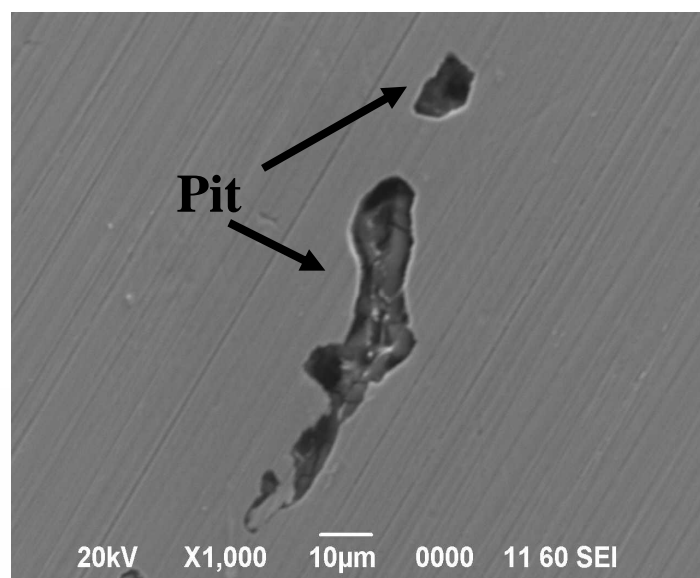


Fig. 3. A surface appearance of $Ni_3(Si,Ti)+2Cr$ after polarization test in 0.1 kmol/m³ neutral NaCl solution at 293 K.

C. Effect of chloride concentration

Fig. 4 shows the relationship between pitting potential (E_{pit}) and a logarithm of chloride concentrations for $Ni_3(Si,Ti) + 2Cr$, $Ni_3(Si,Ti) + 4Al$ (Priyotomo et al.,2014), $Ni_3(Si,Ti)$ (Priyotomo., 2013) and C 276 alloy (Priyotomo., 2013) at 293 K. It was found that the pitting potential of $Ni_3(Si,Ti)+2Cr$ increased linearly with decreasing chloride concentration up to a certain chloride concentration, below which no pitting corrosion occurred as well as the previous works such as $Ni_3(Si,Ti)$ compound, C 276 alloy (Priyotomo.,2013) and $Ni_3(Si,Ti)+4Al$ compound (Priyotomo et al.,2014). Furthermore, the chloride concentration value at which no pitting corrosion occurred was defined as a critical chloride concentration (Cl_{crit}). The presence of the critical chloride concentration means that the parameters of film breakdown rate and repassivation rate are essential as well as the pitting corrosion of the homogenized $Ni_3(Si,Ti)$ compound (Wagle et al.,2011), $Ni_3(Si,Ti)$ compound after a thermomechanical treatment (Priyotomo et al.,2011) and $Ni_3(Si,Ti) + 4Al$ compound after the thermomechanical treatment (Priyotomo et al., 2014). Below the critical chloride concentration, the repassivation rate is superior to the film breakdown rate to show no occurrence of pitting corrosion process. Therefore, it implies that there is no defect on the surface of passive layer.

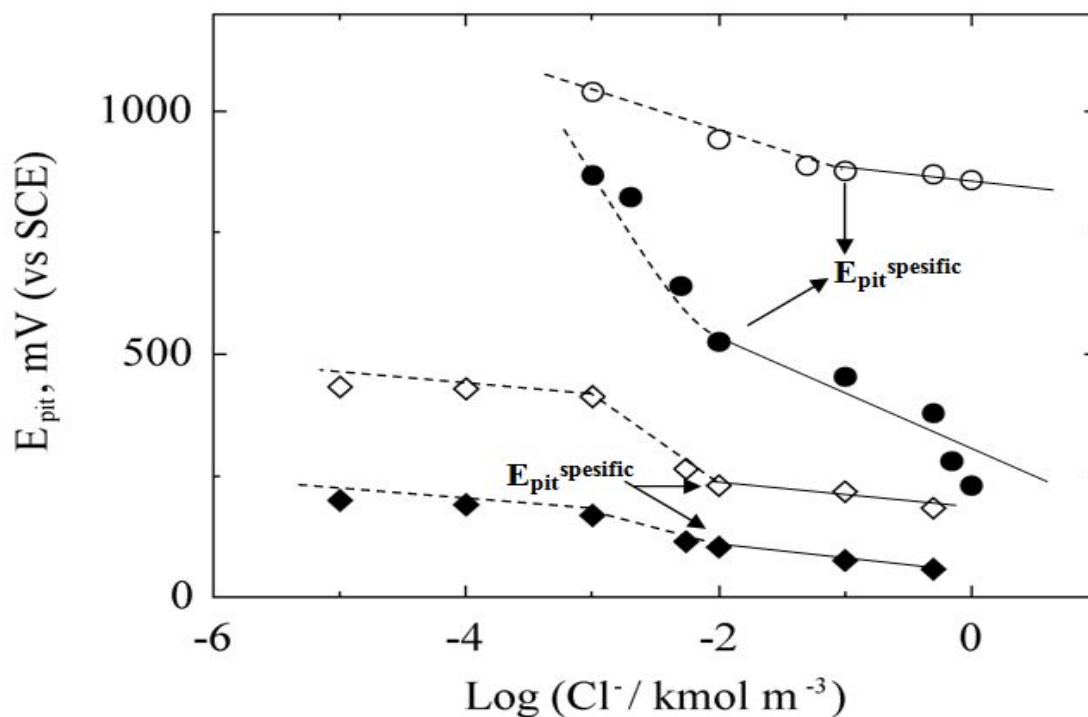


Fig.4 Chloride concentration dependence of pitting potential for \diamond $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$; \blacklozenge $\text{Ni}_3(\text{Si,Ti}) + 4\text{Al}$ (Priyotomo et al.,2014)[16]; \bullet $\text{Ni}_3(\text{Si,Ti})$ (Priyotomo., 2013)[8] and \circ C276 alloy (Priyotomo., 2013)[8] in neutral sodium chloride solutions at 293 K.

From Fig. 4, it was found that the pitting potential values of C276 alloy were the highest compared to among those of $\text{Ni}_3(\text{Si,Ti})$ without (Priyotomo et al., 2013) and with the addition of Al (Priyotomo et al., 2014) and Cr. This proof means that the pitting corrosion resistances were decreased in the order of $\text{C276} > \text{Ni}_3(\text{Si,Ti}) > \text{Ni}_3(\text{Si,Ti}) + 2\text{Cr} > \text{Ni}_3(\text{Si,Ti}) + 4\text{Al}$ in various chloride concentrations. Table 2 shows the relationship between a specific pitting potential ($E_{\text{pit}}^{\text{specific}}$) and critical chloride concentration (Cl_{cri}), where the specific pitting potential is the pitting potential at the critical chloride concentration. From this table, it was found that the specific pitting potential were decreased in the order of $\text{C276} > \text{Ni}_3(\text{Si,Ti}) > \text{Ni}_3(\text{Si,Ti}) + 2\text{Cr} > \text{Ni}_3(\text{Si,Ti}) + 4\text{Al}$, where pitting corrosion could not take place at a potential lower than $E_{\text{pit}}^{\text{specific}}$. Therefore, $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$ has the moderate susceptibility of pitting corrosion compared to $\text{Ni}_3(\text{Si,Ti})$ and C276, where $\text{Ni}_3(\text{Si,Ti}) + 4\text{Al}$ has the worst of pitting corrosion resistance due to the lowest critical chloride concentration.

Table 2. The relationship between the specific pitting potential ($E_{\text{pit}}^{\text{specific}}$) and critical chloride concentration (Cl_{cri}) on $\text{Ni}_3(\text{Si,Ti})$ with and without the addition of Al and Cr and C276 alloy in neutral sodium chloride solution at 293 K.

Compounds	$E_{\text{pit}}^{\text{specific}}$ (mV vs SCE)	Cl_{cri} (kmol/m ³)
$\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$	264	0.0055
$\text{Ni}_3(\text{Si,Ti}) + 4\text{Al}^*$	114.3	0.0055
$\text{Ni}_3(\text{Si,Ti})^{**}$	530	0.01
C276*	889	0.05

* (Priyotomo., 2013) and ** (Priyotomo et al.,2014)

D. Potentiostatic polarization measurement

Fig. 5 shows that the current densities of $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$ intermetallic compound recorded as a function of time for the different constant potential values such as -100 mV, +100 mV, +300 mV, +400 mV and +500 mV vs SCE in 0.1 kmol/m³ neutral NaCl solution at 293 K for the immersion time of 4000 seconds. The current densities at -100mV and +100 mV vs SCE decrease with increasing the time and lies down in the passivity region which started from 50 seconds of time exposure as shown in Fig 6. Furthermore, the current densities as a function of immersion time rise rapidly at 500 seconds, when the various constant applied potentials were higher than the pitting potential (E_{pit}) in 0.1 kmol NaCl solution (E_{pit} : +217 mV vs SCE). On the other hand, by increasing the constant applied potentials above E_{pit} , the current densities increase up to 500 seconds indicating the presence of stable pitting occurred on the surface as well as austenitic stainless steel such as type 304 and type 316L (Ilevbare et al.,2001) and $\text{Ni}_3(\text{Si,Ti}) + 4\text{Al}$ compound (Priyotomo et al, 2014). In addition, the stable propagation of pit was occurred in stable pitting with the growth of macropit (Williams et al.,1994). From 500 seconds of immersion time, at -100 mV and +100 mV vs SCE, the magnitude of current densities for $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$ were lower than those at +300 mV, 400 mV and +500 mV vs SCE, where at 100 mV and +100 mV vs SCE, the current densities of the compound became stable as function of time. Fig 6 shows that the current densities of $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$ below E_{pit} decrease with increasing the immersion time indicating the stable passive condition. Several peaks took place at the stable passive region corresponded to the metastable pit initiation of stainless steel (Ebrahimi et al.,2001).

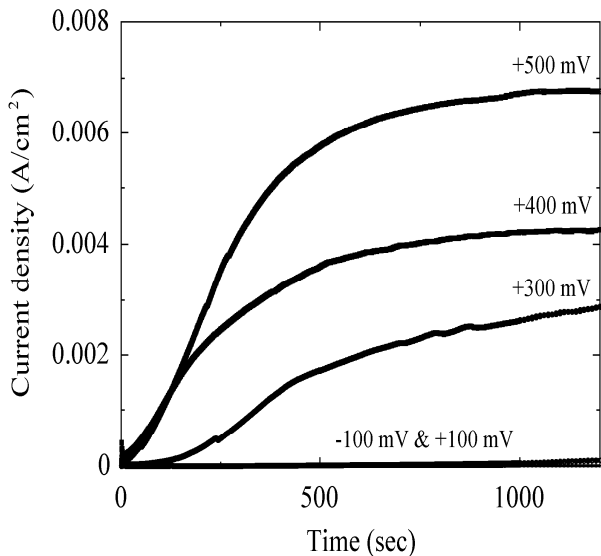


Fig.5 Potentiostatic tests of $\text{Ni}_3(\text{Si,Ti})+2\text{Cr}$ at all constant potentials vs (SCE) as a function of immersion time in 0.1 kmol/m^3 NaCl solution at 293 K

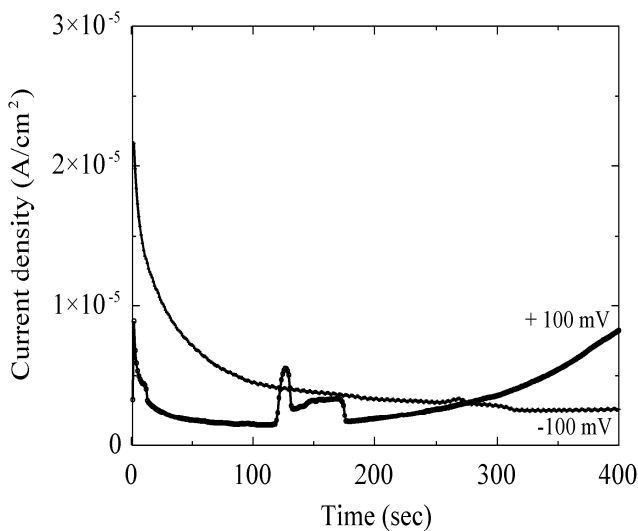


Fig.6 Potentiostatic tests of $\text{Ni}_3(\text{Si,Ti})+2\text{Cr}$ at constant potentials for +100 mV and -100 mV vs (SCE) as a function of immersion time in 0.1 kmol/m^3 solution at 293 K

D. The effect of element distribution to the occurrence of pit

Fig 7 shows the surface appearance of $\text{Ni}_3(\text{Si,Ti})+2\text{Cr}$ after potentiostatic polarization test in 0.1 kmol/m^3 neutral NaCl solution at +300 mV vs SCE. It is difficult to observe the phases such as $\text{L}_{12} + \text{Ni}_{ss}$ mixture phase and L_{12} phase containing the localized attack like pitting corrosion of this compound due to the small size of $\text{L}_{12} + \text{Ni}_{ss}$ mixture phase. On the other hand, in Fig.8, by performing energy dispersive X-ray spectroscopy, the elements of Cr, Si, Ti and O was enriched in the edge or inside of pit, while the average amount of Ni distributed uniformly in the highest weight concentration outside pits. The presence of oxygen with high concentration level in the edge or inside of pit could be took place, where the hydrolysis process of the dissolved metal ion took place to produce free acid containing the cation of hydrogen inside pit and the hydroxide in adjacent pit (Trethewey et

al.,1988). Therefore, it implies that the presence of oxygen level in high concentration indicate the occurrence of pitting process.

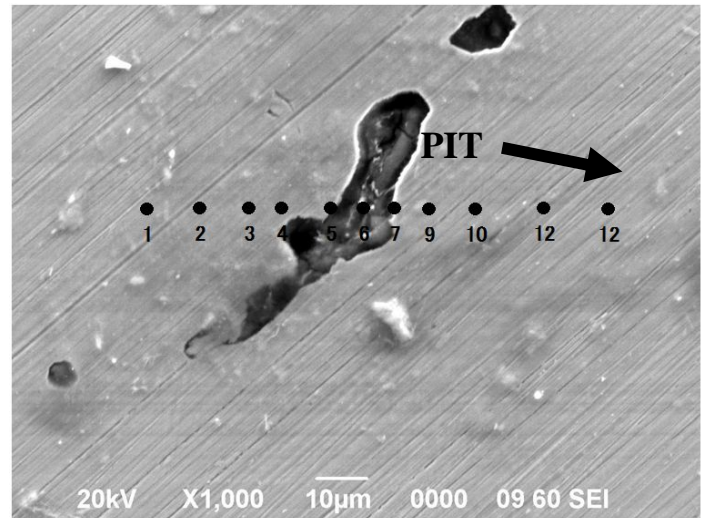


Fig. 7. The surface appearance of $\text{Ni}_3(\text{Si,Ti})+2\text{Cr}$ after the potentiostatic polarization test in 0.1 kmol/m^3 neutral NaCl solution at constant potential of +300 mV vs SCE.

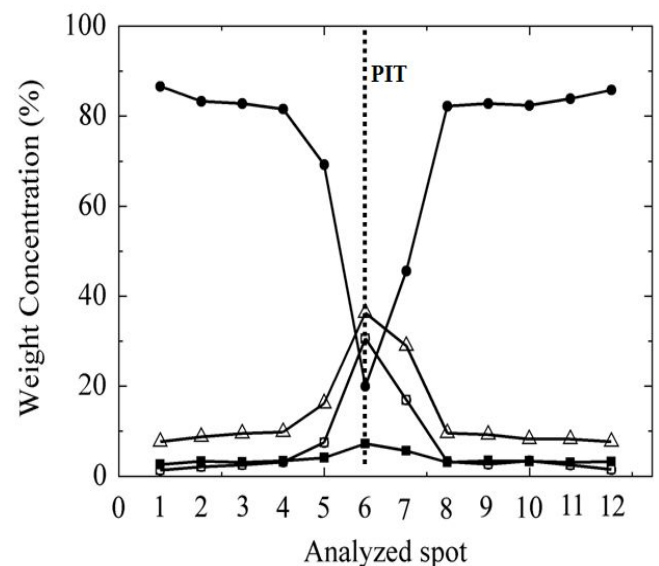


Fig.8. Plot of the measured weight concentrations of \blacksquare Si, \triangle Ti, \bullet Ni, \circ Cr and \square O for all spots analyzed on sample of $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$ as shown in Fig.7.

IV. CONCLUSION

The pitting corrosion behavior of $\text{Ni}_3(\text{Si,Ti})+2\text{Cr}$ intermetallic compound in various neutral sodium chloride solution has been investigated and compared with that of $\text{Ni}_3(\text{Si,Ti})$ with and without Al and C276 alloy. The following things were obtained as follows.

1. The pitting potential of $\text{Ni}_3(\text{Si,Ti}) + 2\text{Cr}$ decreased with increasing chloride concentration above the critical chloride concentration, below which no pitting corrosion took place as well as those of $\text{Ni}_3(\text{Si,Ti})$ with and without Al and C276 alloy.
2. The specific pitting potentials and the pitting potentials were decreased in the order of C276 alloy > $\text{Ni}_3(\text{Si,Ti}) > \text{Ni}_3(\text{Si,Ti}) + 2\text{Cr} > \text{Ni}_3(\text{Si,Ti}) + 4\text{Al}$

3. The pitting corrosion resistances were decreased in the order of C276 alloy > Ni₃(Si,Ti) > Ni₃(Si,Ti) + 2Cr > Ni₃(Si,Ti) + 4Al in various chloride concentrations.
4. The presence of oxygen level in high concentration indicates the occurrence of pit.

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